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## Magnesia Bricks for Lining Cement Kilns.

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IN connection with the article by M. Felix Krémer published in CEMENT AND LIME MANUFACTURE for November, 1939, it may be of interest to add the following results of laboratory tests and experience in practice.

The laboratory tests, in which the composition of the raw materials fluctuated considerably, aimed at examining the reaction of these raw materials to various refractory bricks, particularly high-alumina, magnesia, and clay bricks (with about 40 per cent.  $\text{Al}_2\text{O}_3$ ). The tests were made with raw meals moulded to 1 in. by 3 in. briquettes which were placed on the trial bricks and burnt in an oil-fired laboratory furnace. The burning conditions were always the same, that is, the briquettes, lying with their base on the brick, were heated to 1,450 deg. C., maintained at that temperature for twenty minutes, and then cooled. As only the quantity of the fusion formed between clinker and brick and its penetration were of interest, it was of small importance if the cooling conditions differed from those prevailing in the kiln.

Chemical composition (lime saturation value, silica and iron moduli, and ash content), and also the fineness of the raw meal, were systematically varied ; but with these factors extremely fluctuating no reaction could be found on the surface of magnesia bricks. The raw meal briquettes, after having been burnt to clinker on the trial bricks, could be freely moved ; there was not the slightest sticking. In the case of raw meals which were burnt to clinker under testing conditions the clay bricks showed strong adherence, and much molten material was formed between the clinker and the brick, and this penetrated into the clinker as well as into the brick. The higher the ash content and the lower the silica and iron moduli and the lime saturation value the stronger was this effect.

With high-alumina bricks (60 to 70 per cent.  $\text{Al}_2\text{O}_3$ ) the reaction between the clinker and brick started at higher temperatures, and although less molten material was produced it stuck much faster to the surface of the brick than in the case of clay bricks under the same testing conditions.

These results confirmed the conclusions which can be drawn from the phase-rule diagrams between  $\text{Al}_2\text{O}_3$  and the components of cement clinker. Only the action of the ashes seems to be an exception ; this action upon the brickwork is stronger than would correspond to the modification of the chemical composition. This was also confirmed in rotary kilns ; when the ash content of the coal is small and uniform, the life of alumina bricks in the clinkering zone is considerably prolonged. In an oil-fired rotary kiln, where there is practically no ash and where the clinkering zone was lined with clay bricks only, after  $1\frac{1}{2}$  years' running of the kiln the lining was in good condition although the output of the kiln was always the highest possible. Of the original brick thickness of 15 cm., about 8 cm. was intact, 2 to 3 cm. was the thickness of the partly-molten reaction product, and 4 to 5 cm. was a somewhat loose clinker coating. Whenever the chemical composition of the raw material can be kept definitely constant a good coating in the clinker zone is a great advantage, as it protects the underlying brickwork and thereby favourably influences the heat insulation. If the kiln is carefully fired the thickness of the coating can also be kept constant. With raw materials having unusual properties and a chemical composition subject to fluctuation it is possible that the coating may become too thick and thus troublesome. In such cases it is far more advantageous to use high-magnesia bricks so that the reaction between the clinkering raw meal and the kiln lining occurs only at much higher temperatures. Under normal firing conditions the high-magnesia brickwork in rotary kilns is not sensitive to fluctuations of the chemical composition and ash content ; however, due to its greater heat conductivity, it is practicable to have a strong insulating layer between the brick and the kiln shell. If the insulating layer is strong enough and the burning temperature sufficiently high, it is possible for a coating to be burnt on high magnesia bricks.

Simultaneously with the laboratory tests I watched for several years the behaviour of high-magnesia bricks, alumina bricks, and clay bricks in automatic shaft kilns. Here the sticking of the material to the lining in the clinker zone is in all circumstances a disadvantage, leading to failures in output and quality. Experiments with the most expensive magnesia bricks have not led to satisfactory results. Not only in the upper part of the kiln was the lining destroyed—possibly due to the moisture in the mixture of raw meal and coal which is fed into the kiln in the form of small briquettes—but it was also destroyed in the clinkering zone. Even with a careful and uniform preparation of the raw material it happens in shaft kilns that the clinkering zone moves up and down, so that all the bricks are sometimes subjected to the action of steam generated from the briquettes. Then the lining cracks, becomes loose in the brickwork, and falls to pieces.

These conclusions were obtained from experiences and tests made about three years ago. M. Krémer in his article intimated that nowadays it is possible

to manufacture magnesia bricks which are insensitive to the action of steam. In this connection, however, it is remarkable that even to-day a special dry laying is specified for high-magnesia bricks, which would not appear to be necessary were the bricks absolutely resistant to moisture.

In shaft kilns alumina bricks with 61 per cent.  $\text{Al}_2\text{O}_3$  behaved in a less favourable manner; while fusion occurred rather seldom, nevertheless if it occurred it stuck so fast to the brick that it was very difficult to remove it. This experience is in conformity with test results in an oil-fired laboratory furnace. For this reason it is, in special circumstances, more advantageous to use clay bricks, where subsequently occurring fusion (clinging) is easily removable. Naturally this varies with different kinds of materials, the size of the raw meal-coal briquettes, and the moisture content. The plasticity of the raw material also influences the possibilities of reaction between the clinker and the kiln-lining. Hence the brick most favourable for local conditions must be selected.

In up-to-date automatic shaft-kilns, where the kiln is fed with very small briquettes which are often blended with a small percentage of fine grains of clinker, and burnt with a fuel consumption of only 900 calories per kg. of clinker for highest grade cements, the maintenance of a uniform chemical composition is much more important than in the case of rotary kilns. If this state could be reached, an absolutely even clinker burning without clinging to the lining would be possible even with high-alumina bricks in shaft kilns. However, notwithstanding modern methods of preparation and homogenising, this condition is not always feasible in practice. For this reason a kiln lining perfectly insensitive to the action of moisture and to minor fluctuations of the chemical composition and coal charge would be very welcome, especially for shaft kilns.

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## Influence of the Heat-Treatment of Portland Cement Clinker on the Properties of the Cement.\*

THE following is an abstract of a paper by Mr. T. W. Parker, of the Building Research Station, published in the *Journal of the Society of Chemical Industry* for June, 1939.

An investigation has been made at the Building Research Station of the Department of Scientific and Industrial Research into the effect on the properties of the resultant cement of the manner in which Portland cement clinker is cooled from the burning temperature. The need for such an investigation had become apparent from the results of phase equilibrium studies on the four major oxide constituents ( $\text{CaO}$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{SiO}_2$ ,  $\text{Fe}_2\text{O}_3$ ) of Portland cement<sup>1</sup> and the conceptions of the constitution of cement clinker which have arisen from this work.<sup>2</sup> The basic hypothesis is that during manufacture the clinker, apart from some small residue of uncombined lime, reaches a state of chemical equilibrium in the burning zone of the rotary kiln, but that equilibrium is not maintained on subsequent cooling.

At the hot zone the clinker consists of a mixture of solid crystalline silicates and a molten liquid containing all the alumina and iron oxide, together with lime, a small amount of silica, and the greater part of the remaining minor components. On subsequent cooling, the assumption is made that the solid crystals and the molten liquid behave as independent systems, there being no reaction between the two such as would be necessary if chemical equilibrium were to be maintained. The independent cooling of the liquid may result in it being converted into a glass, if the cooling is sufficiently rapid; or it may be converted into a crystalline mixture containing  $3\text{CaO}, \text{Al}_2\text{O}_3$ ,  $4\text{CaO}, \text{Al}_2\text{O}_3, \text{Fe}_2\text{O}_3$ , possibly together with  $5\text{CaO}, 3\text{Al}_2\text{O}_3$  or  $\text{CaO}$  (depending on the composition of the liquid), if the cooling is sufficiently slow; or it may be converted into an intermediate stage in which glass with one or more of the crystalline compounds mentioned above may be present.

It is possible to derive equations for calculating the compound compositions of clinkers for either of the two extreme cases,<sup>3</sup> but the intermediate condition is less amenable to theoretical treatment in the present stage of research in this field. L. A. Dahl<sup>4</sup> has developed formulae based on phase crystallisation courses of the liquid for intermediate conditions, but the principles of his method may

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<sup>1</sup> F. M. LEA and T. W. PARKER, *Phil. Trans.*, 1934, 234, No. 731.

<sup>2</sup> LEA, *Cement*, 1935, 2, No. 8.

<sup>3</sup> LEA and PARKER, *Building Res. Tech. Paper* 16, 1934.

<sup>4</sup> *Rock Products*, 1938, 41, No. 9, 48 et seq.

not hold good under the temperature conditions in the manufacture of clinker in the rotary kiln.<sup>5</sup>

The importance of a reliable means of determining the compound content lies in the possibility of predicting the physical properties of a cement from its chemical composition and fineness alone, instead of from performance tests. It is therefore desirable to know whether the physical properties of the cement are changed appreciably if the clinker contains glass or if it is completely crystalline. If there is no change in physical properties it is obviously a matter of indifference which method of calculation is used ; if, on the other hand, there are differences in physical properties it is desirable to know the condition which gives the most favourable results.

Recent investigations<sup>6,7</sup> have shown that the assumption above may need some modification to account for the compound formed by the alkalis and the possibility that completely suspended equilibrium may not always occur, but these factors were not seriously involved in the present work.

The programme of the present investigation in its simplest aspects was to prepare a few clinker compositions ; to control the heat-treatment so that direct comparison could be made of products of identical composition so cooled as to be in complete crystalline equilibrium and quenched so that the molten part was cooled to a glass ; to prepare cements from these clinkers, and to determine the properties of the cements. However, it was not found possible to retain the programme in this very simple form. Other factors are involved which influence the properties of the cement and must therefore be taken into account. For example, small variations in the quantity of gypsum added to control the setting time have an influence on the strength of the cement and on the quantity of mixing water necessary to make a paste of normal consistence. It was therefore necessary to standardise a procedure for gypsum additions in a manner described later. The fineness of the cement also affects its properties ; tests were accordingly made at two different finenesses in all cases. The possibility of magnesia and alkalis having an influence on the properties had also to be taken into account.

In the final programme, therefore, it was decided to prepare two basic compositions, differing only in the ratio of  $3\text{CaO}, \text{SiO}_2$  to  $2\text{CaO}, \text{SiO}_2$ , but having in both cases as nearly similar as possible contents of alumina and of ferric oxide. In order to ascertain the influence of magnesia and alkalis, additional samples were made from each composition by adding to them a definite quantity of these oxides. Samples from the resulting four compositions were prepared in both the quenched and fully crystalline state, producing thereby eight samples, and this number was multiplied further by testing most of the samples at two fine-

<sup>5</sup> T. W. PARKER, Discussion on Bogue's paper.<sup>6</sup>

<sup>6</sup> R. H. BOGUE, "Constitution of Portland Cement Clinker," Symposium on the Chemistry of Cements, Stockholm, 1938. H. INSLEY and H. F. McMURDIE, *J. Res. Nat. Bur. Stand.*, 1938, 20, 173.

<sup>7</sup> INSLEY, *J. Res. Nat. Bur. Stand.*, 1936, 17, 353. T. W. PARKER and R. W. NURSE, *J. Soc. Chem. Ind.*, 1939, 58, 255-261.

<sup>8</sup> H. INSLEY, E. P. FLINT, E. S. NEWMAN, and J. A. SWENSON, *J. Res. Nat. Bur. Stand.*, 1938, 21, 355.

nesses, one being about that of normal Portland cements, the other about that of rapid-hardening Portland cements.

Two other compositions were also tested, one being derived from a commercial wet-kiln clinker and the other from a dry-kiln clinker. The sub-division of these into samples was rather different from those above. Cements were prepared at two finenesses from the clinker (i) as received, (ii) after reheating and quenching, and (iii) after reheating and crystallising fully. Further samples were obtained and tested at one fineness only by sieving the clinker to separate the coarse, medium, and small grain sizes respectively. These were made to ascertain whether the clinker was uniform in properties throughout. Finally two additional samples were obtained of the dry-kiln clinker, the one being the normally cooled product and the other material collected at the same time by quenching clinker direct from the hot zone of the kiln. Larger samples than could be made in the laboratory were thus obtained.

The details of the samples are set out in Table III ; in all, 31 samples were tested. Only relatively small quantities of each sample could be prepared in reasonable time in the laboratory, and it became necessary, therefore, to use small-scale methods for testing their properties. Details of these methods are given later.

### Experimental.

The synthetic clinkers were prepared from samples of calcium carbonate, alumina, silica, and ferric oxide of high purity. These materials, in the desired proportions, were mixed with water and compressed into briquettes, each weighing about 500 g., which were placed on platinum sheets and fired in a gas furnace. After one hour at 1,200 deg. they were remixed with water, again compressed, and fired for three hours at 1,400 deg. The resulting clinker was ground and separated into two portions. One portion was heated for one hour at 1,400 deg. in an electric muffle and then quenched in water ; the other portion, after one hour at 1,400 deg., was cooled during one hour to 1,280 deg. and maintained at this temperature for two hours or more. If, however, an annealed melt showed signs of dusting on subsequent cooling it was quenched in water from the lowest temperature at which this could be prevented.

These processes produced the glassy and the crystalline clinkers respectively. The commercial clinkers were ground and heat-treated in a manner similar to that used for the final stage of those prepared in the laboratory.

The next process was that of grinding and introduction of gypsum to control the set. For this purpose, preliminary trials were made with each clinker, grinding small samples in a ball mill capable of dealing with 30 g. of material with varying amounts of gypsum.

The small 30-g. samples were tested for the amount of water required to give normal consistence, for setting time, and for mortar strength at 7 days.

It was found that an SO<sub>3</sub> content of 2½ per cent. was suitable for all low-lime cements, and 2 per cent. for the high-lime cements. The SO<sub>3</sub> contents were

chosen to give normal setting times, and strengths as near as possible to the optimum values.

Following the trials, the remainder of the batch was ground in a larger ball mill. Tests similar to those described above were made for the batches to be examined at the second fineness. The batches were then tested for consistence, setting time, soundness, strength, chemical resistance, shrinkage, and (in one case) heat of hydration. For all these tests it was necessary to use the small-scale methods described below.

*Consistence.*—Several attempts were made to devise a mechanical small-scale method to determine the amount of water necessary for mixing to a standard plastic consistence. Check tests on samples of commercial cements showed, however, that none of them was as accurate or reproducible as that given by estimation by the gauger. Consequently the latter method was used throughout. The same gauger carried out all the work. The water content used for mortars was chosen to give a consistence corresponding with that obtained with 11 per cent. of water on 1 : 3 standard sand mortars.

*Setting time.*—A small cylindrical brass mould was used, of dimensions 15 mm. diameter by 15 mm. depth. Tests were made on this with commercial cements the setting time of which had been determined in the ordinary way, and data were obtained of the depth of penetration of the normal Vicat needle at different times. These data were plotted as a graph, for each cement. At the time corresponding with the initial set, as determined in the ordinary way, the needle penetrated to a depth of 12 mm. and at a time corresponding with final set the penetration was 0 mm. The initial set could be estimated to within  $\pm$  10 minutes by the time elapsing up to the 12-mm. penetration, and the final set to within  $\pm$  10 minutes by the time elapsing to zero penetration.

*Soundness.*—Soundness was determined in the setting time apparatus, a modified form of the latter being used. The mould size was the same, but the mould ring was split and pointers were attached of the same length as in the ordinary Le Chatelier apparatus for testing soundness. After carrying out the setting time test, soundness tests were made using the same procedure as in the British Standard full-scale method. The specimens were stored for 24 hours in water at 18 deg., measured, then steamed for 3 hours, and again measured.

*Strength.*—Strength tests were carried out on 1 : 3 sand mortars of plastic consistence, the specimens being cubes of  $\frac{1}{2}$  in. side, tested in compression. Preliminary trials showed that very great care was required in the preparation and testing of these small specimens, especially to reduce the spread of results from six cubes tested at the same age. The following was the process finally adopted. The sand was graded to a size all passing the British Standard 52-mesh sieve, but retained on the British Standard 72-mesh sieve, and was washed thoroughly before use. Distilled water was used for mixing.

Mix proportions of 1 cement : 3 sand by weight were used in all tests. The amounts required for a set of six cubes were about 7 g. of cement and 21 g. of sand. The actual quantities were weighed out to the nearest 0.01 g. and the

amount of water required for mixing was added through a small burette to the nearest 0.05 ml. Mixing was carried out on a small, smooth glass plate with a palette knife, and the same gauger performed all the necessary work. The mortars were filled into the moulds in two operations, tamping with a  $\frac{1}{4}$ -in. square steel rod at each stage until a layer of water began to appear, and finally smoothing with the palette knife. At the end of the operation, the top of the specimen was covered with a glass plate and the specimen stored in moist air (18 deg. and above 90 per cent. relative humidity) for 1 day. The cubes were then removed from the moulds, taking care to avoid damage to edges, etc., and subsequently stored in water at 18 deg. until required. Six cubes were made for test at each age and in general four ages of test, 1, 3, 7, and 28 days, were used. The cubes were distributed between the different test ages so that the cubes made at one gauging were not all tested at one age, but were spread over the four ages. This spreading was adopted to minimise personal error in gauging.

Compression tests were carried out on a transverse testing machine adapted for the purpose. The load was transmitted through a ball seating, special care being taken to obtain accurate centering, and was applied at the rate of 300 lb. per minute. The tests were made within 20 minutes of the exact test age required. The average probable errors were 9.8, 9.5, 9.2, and 7.0 per cent. of the means at 1, 3, 7, and 28 days respectively.

The results obtained by this method were correlated for several commercial cements with those obtained using cubes of 1 : 3 mortars having 50 sq. cm. sides. If  $S_L$  represents the strength at any age in lb. per sq. in. of 1 : 3 standard sand mortars (water/cement = 0.50), hand-compacted, tested on 50 sq. cm. side cubes, and  $S_s$  represents the corresponding small-scale value, then  $S_L = 1.37S_s - 210$ . The constant 210 indicates a diversion from true proportionality at low strengths (since such a correlation must hold for  $S_L = S_s = 0$ ) but the relation holds for all strengths higher than 300 lb. per sq. in.

*Resistance to Attack by Solutions containing Sulphates.*—Tests of the resistance to attack by sulphate solutions were made on 1 : 3 cement : sand mortars of a plastic consistence similar to that for the strength tests. Cylindrical specimens 1 in. long and  $\frac{1}{2}$  in. in diameter were prepared and small steel balls were cemented into the ends. These specimens were stored in moist air for 1 day, then in water for 27 days. At the end of this storage period two specimens of each cement under test were completely immersed in (i) 5 per cent. magnesium sulphate solution, (ii) 5 per cent. sodium sulphate solution, and (iii) distilled water. The length between the balls was measured, by means of a micrometer capable of reading by estimation to 0.0001 in., at the start of the test and at intervals thereafter.

Expansion in the sulphate solutions was more rapid than with the larger-scale similar tests which have been made at this Station on 5 in.  $\times$  1 in. rods of various cement mortars. Preliminary tests showed that when the initial storage period was only 7 days the expansion was even more rapid. It was intended, however, not that the small-scale tests should be directly related to the larger scale, but that they should be used only for comparison amongst themselves.

Larger-scale tests were made in addition on one of the commercial clinkers to demonstrate that the small-scale results were in the same order as would have been obtained on the larger scale.

**Shrinkage.**—Small-scale volume shrinkage tests were made on 1-in. cubes of 1:3 mortars. The method devised for this has already been described.<sup>9</sup>

**Heat of Hydration.**—Tests were made with a small-scale method of determining the heat of dissolution of the neat and the hydrated cements according to the usual method.<sup>10</sup> The results were, however, rather variable and it was considered that the data obtained were not reliable. Accordingly one test was made on a larger sample with the adiabatic calorimeter developed at the Station.<sup>11</sup>

**Fineness.**—The finenesses of the samples were determined on a Wagner turbidimeter, modified in a way already described.<sup>12</sup>

### Results.

The results in detail are given in tabulated form in Tables II to VII. The serial numbers attached to the cements are those given for identification purposes. The letter "X" after the serial number indicates that the cement was fully crystalline\* and "G" indicates that it was quenched to produce a glassy type. In addition, for the commercial clinkers, "O" indicates the cement as received, "L," "M," and "S" represent samples of large-grained, medium-grained, and small-grained clinker pieces respectively.

Table I gives a general description of the cements, including the observations on the microscopic examination of the clinker, the amount of SO<sub>3</sub> added for setting control, and the time required for grinding to the required specific surface. The method used for microscopic examination was to take small powder samples after each separate quenching or annealing operation, and to examine these by transmitted light. Larger clinker pieces were also sampled from each complete batch and polished for examination by reflected light. The observations in Table I refer to the examination by reflected light, after etching the specimen with water followed by a dilute solution of nitric acid in alcohol.

Table II gives the chemical analyses of the cements and Table III the calculated compound contents. These have been calculated in three ways: by the Bogue method and by the two methods developed on the assumption of frozen equilibria.<sup>3</sup> The figures given as glass contents do not include magnesia and alkalis. It will be clear that the method which involves the calculation of the alumina and ferric oxide compounds, etc., as a glass does not apply to "X" melts, although calculated values have been given. Similarly the calculations for complete crystallisation do not apply to "G" melts. However, it was found to be very difficult to prepare "X" and "G" melts of identical oxide composition, and the slight differences are sometimes magnified in the calculation for compound content.

\* R. W. NURSE, J.S.C.I., 1939, 58, 37.

<sup>10</sup> H. WOODS, H. N. STEINOUR, and H. R. STARKE, *Ind. Eng. Chem.*, 1932, 24, 1207.

<sup>11</sup> N. DAVEY, *Concrete and Constr. Eng.*, 1931, 26, 572.

<sup>12</sup> T. W. PARKER and R. W. NURSE, J.S.C.I., 1938, 57, 436.

\* The "X" cements are assumed to be at full crystalline equilibrium, not at fully crystalline frozen equilibrium.

TABLE I.—PHYSICAL PROPERTIES OF CLINKERS.

Cement No.	Type	Heat treatment	Microscopic examination	Optimum SO <sub>3</sub> , %	Time of grinding, min.	Specific surface, sq. cm./g.
14 X	Low-lime	Annealed	Well crystallised, but interstitial crystals not very large. Some tendency to dust	2.5	73	2250
14 G	"	Quenched	Most of interstitial material clear glass. A few centres of crystallisation in the glass	2.5	150	2290
15 X	Low-lime with MgO and alkalis	Annealed	Well crystallised, interstitial crystals fairly large	2.5	120	2340
15 G	Low-lime with MgO and alkalis	Quenched	Most of interstitial material glass. A little crystallisation of the glass in places	2.5	125	2300
16 X	Low-lime†	Annealed	Well crystallised, interstitial crystals fairly large	2.5	60	1410
16 G	"	Quenched	Much crystallisation of the interstitial glass. In places approaches the fully crystalline condition	2.5	95	1525
17 X	High-lime	Annealed	Crystalline. Interstitial crystals quite large	2.0	A. 50 B. 65	1590 1930
17 G	"	Quenched	Most of interstitial glass. Glass contains some centres of crystallisation	2.0	A. 65 B. 90	1700 1970
18 X	High-lime with MgO and alkalis	Annealed	Crystalline. Very large interstitial crystals	2.0	A. 60 B. 80	1650 2010
18 G	High-lime, with MgO and alkalis	Quenched	Very little crystallisation of the glass	2.0	A. 75 B. 100	1630 2000
19 X	Wet-kiln clinker	Annealed	Crystalline. Large interstitial crystals	2.0	A. 110 B. 160	1580 2020
19 G	"	Quenched	Interstitial material entirely glass	2.0	A. 125 B. 160	1590 2020
19 O	"	Rotary kiln	Interstitial crystallisation, generally small crystals. An intermediate condition	2.0	A. 115 B. 137	1640 1910
19 L	"	"	As 19 O	2.0	110	1630
19 M	"	"	"	2.0	110	1620
19 S	"	"	"	2.0	105	1590
20 X	Dry-kiln clinker	Annealed	Well crystallised. Large interstitial crystals, well developed. MgO seen	2.0	95	1610
20 G	"	Quenched	Interstitial material entirely glass	2.0	125	1560
20 O	"	Rotary kiln	Appears entirely crystalline, but interstitial crystals small and intergrown. No MgO seen	2.0	120	1550
20 L	"	"	As 20 O	2.0	100	1560
20 M	"	"	"	2.0	95	1620
20 S	"	"	"	2.0	145	1580
24	"	Rotary kiln attempted quenching	Considerable variation in condition from crust to centre of clinker pieces. Some entirely glassy, some similar to 20 O. Much of the clinker has glassy interstitial material with dendritic crystal growths	2.0	135	1760
25	"	Rotary kiln	As 20 O	2.0	125	1740

† Composition No. 16 was intended to be a repeat of composition No. 14, but more coarsely ground. There is, however, a considerable difference in composition.

TABLE II.—ANALYSES OF CLINKERS.

Clinker No.	CaO	Free CaO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	FeO	MgO	K <sub>2</sub> O	Na <sub>2</sub> O	SO <sub>3</sub>	TiO <sub>2</sub>
14 X	64.96	0.4	22.88	8.53	2.73	0.32	nil	nil	nil	nil	nil
14 G	65.08	0.4	22.53	8.38	3.00	0.20	—	—	—	—	—
15 X	63.96	0.3	22.26	7.78	2.93	0.55	1.97	0.36	0.75	—	—
15 G	63.78	0.3	22.04	8.20	3.41	0.22	1.89	0.11	0.24	0.22	—
16 X	65.47	0.0	22.48	8.00	3.18	nil	nil	nil	nil	—	—
16 G	65.66	0.0	22.42	8.03	3.22	—	—	—	—	—	—
17 X	67.42	0.6	20.86	8.08	3.33	—	—	—	—	—	—
17 G	67.22	0.6	20.86	8.26	3.36	—	—	—	—	—	—
18 X	66.00	0.2	20.22	8.06	3.32	trace	1.93	0.13	0.27	—	—
18 G	65.97	0.2	20.36	8.04	3.30	—	2.00	0.16	0.34	—	—
19 O	64.81	0.6	22.40	5.70	2.63	0.13	2.51	0.71	0.31	0.88	0.23
19 X	64.88	0.0	22.50	5.73	2.78	0.01	2.52	0.49	0.25	0.86	0.23
19 G	64.92	0.1	22.45	5.48	2.73	0.07	2.67	0.49	0.27	0.56	0.23
19 L	64.64	0.9	22.46	5.44	2.49	nil	2.61	0.65	0.45	0.48	0.25
19 M	64.60	1.0	22.16	5.41	2.50	0.05	2.74	0.71	0.41	0.38	0.25
19 S	65.09	0.8	21.90	5.86	2.53	nil	2.42	0.56	0.39	0.52	0.25
20 O	66.01	0.4	21.56	6.26	2.44	—	2.43	0.25	0.19	0.38	0.23
20 X	66.10	0.5	21.65	6.38	2.55	—	2.42	0.16	0.17	0.05	0.25
20 G	65.84	0.2	21.54	6.00	2.61	—	2.27	0.16	0.14	nil	0.23
20 L	65.88	0.3	21.15	6.31	2.41	trace	2.54	0.32	0.24	0.74	0.23
20 M	65.55	0.3	21.19	6.67	2.53	nil	2.52	0.21	0.20	0.58	0.23
20 S	66.22	0.3	21.76	5.97	2.29	—	2.39	0.26	0.22	0.27	0.22
24*	65.08	0.3	21.41	5.65	2.33	0.02	2.01	0.24	0.25	2.47*	0.19
25*	65.77	0.4	21.38	5.63	2.26	nil	2.06	0.24	0.22	2.37*	—

\* Analysis on clinker + 2% SO<sub>3</sub> added as gypsum.

TABLE III.—COMPOUND CONTENT OF CLINKERS.

Clinker No.	Bogue calculation				Lea and Parker <sup>1</sup>			Lea and Parker <sup>2</sup>				Lea and Parker <sup>2</sup>
	C <sub>2</sub> S	C <sub>3</sub> S	C <sub>4</sub> A	C <sub>4</sub> AF	C <sub>2</sub> S	C <sub>3</sub> S	Glass	C <sub>2</sub> S	C <sub>3</sub> S	C <sub>4</sub> AF	C <sub>4</sub> A	C <sub>2</sub> A <sub>2</sub>
14 X	28	45	19	7	37	31	30	37	38	7	11	6
14 G	32	41	18	8	39	29	31	39	35	3	11	5
15 X	34	38	17	7	42	26	28	42	32	7	10	5
15 G	31	40	16	10	37	29	31	37	35	10	11	4
16 X	37	37	16	10	43	26	31	43	32	10	11	4
16 G	38	36	16	10	44	25	31	44	31	10	11	4
17 X	54	19	16	10	60	8	31	60	15	10	11	4
17 G	52	21	15	10	58	10	32	58	16	10	11	4
18 X	55	17	16	10	60	6	31	60	12	10	11	4
18 G	54	18	16	10	59	7	31	59	13	10	11	4
19 O	47	29	11	8	50	22	22	50	26	8	8	2
19 X	48	28	11	8	51	22	23	51	26	8	8	2
19 G	51	26	10	8	53	20	22	53	24	8	8	2
19 L	47	29	10	8	51	22	22	50	27	8	8	2
19 M	45	28	10	7	52	20	21	52	24	7	8	2
19 S	51	25	11	8	54	17	23	54	22	8	8	2
20 O	57	19	12	7	61	11	24	61	16	7	9	3
20 X	56	20	13	8	60	12	24	60	16	8	9	3
20 G	59	17	11	8	63	9	23	63	14	8	8	3
20 L	58	17	13	7	63	8	24	63	13	7	9	3
20 M	55	20	13	8	60	11	25	60	16	8	9	3
20 S	59	18	12	7	63	10	23	63	15	7	8	3
24	53	22	11	7	56	14	22	56	19	7	8	3
25	56	19	11	7	60	12	22	60	16	7	8	3

TABLE IV.—SOUNDNESS, SETTING TIME, AND STRENGTH OF CEMENTS.

Cement No.	Composition	Specific surface sq. cm./g.	SO <sub>3</sub> %	Water for normal consistency (heat), %	Le Chatelier test	Setting time hr. min.	Initial expansion mm.	Final expansion mm.	Water for normal consistency (mortar), %	Compressive strength, lb./sq. in.						
										Cold	Hot	hr. min.	hr. min.	1 day	3 days	7 days
14 X	L.L.	3250	2.5	24.5	31	1/2	0	36	1	9	15.5	1	872	1761	2470	3960
14 G	L.L.	2280	2.5	22.0	1	1	0	38	1	12	12.5	1	872	1461	2016	2730
15 X	L.L.M.A.	2340	2.5	28.5	1	1	0	38	1	34	15.5	1	499	1524	2279	3807
15 G	L.L.M.A.	2300	2.5	22.0	1	1	0	32	1	32	13.0	1	821	1524	2279	3807
16 X	L.L.	1410	2.5	24.0	3	nil	1	27	2	45	14.1	1	448	829	1365	2433
16 G	L.L.	1523	2.5	22.0	1	1	0	57	3	40	14.0	1	337	896	1489	2553
17 X	H.L.	1590	2.0	21.0	1	1	0	50	3	30	13.1	1	445	1553	2352	3164
17 G	H.L.	1700	2.0	21.0	1	1	0	50	3	10	13.1	1	651	1508	2511	3847
18 X	H.L.M.A.	1650	2.0	22.0	1	1	0	18	2	54	13.5	1	386	1243	2121	3014
18 G	H.L.M.A.	1630	2.0	21.0	1	1	0	50	3	20	13.1	1	553	1045	1895	3745
17 X	H.L.	1980	2.0	22.0	1	1	0	28	2	59	13.5	1	742	1834	2449	3375
17 G	H.L.	1970	2.0	21.0	2	21	0	23	3	50	13.1	1	629	1599	2397	3868
18 X	H.L.M.A.	2010	2.0	22.0	1	1	0	50	3	5	13.5	1	617	1778	2738	3432
18 G	H.L.M.A.	2000	2.0	21.0	1	1	0	50	3	5	13.1	1	650	1251	2153	4001
19 O	C.	1640	2.0	22.0	1	1	0	20	3	42	13.6	1	366	1403	2263	3375
19 X	C.	1580	2.0	21.0	1	1	0	37	3	10	13.6	1	508	1513	2138	3310
19 G	C.	1590	2.0	21.0	1	1	0	28	4	20	13.6	1	444	1368	1861	3365
19 O	C.	1910	2.0	21.0	1	1	0	32	3	28	12.5	1	530	1656	2570	3361
19 X	C.	2020	2.0	22.0	1	1	0	30	2	36	13.8	1	685	1420	2393	3339
19 G	C.	2020	2.0	21.0	1	1	0	39	3	29	13.5	1	679	1679	2460	3374
19 L	C.	1630	2.0	21.0	1	1	0	25	3	27	13.1	1	449	1512	2151	3187
19 M	C.	1620	2.0	22.0	1	2	1	21	3	30	13.6	1	390	1281	2171	3387
19 S	C.	1590	2.0	21.0	1	2	16	34	13.1	423	1513	1	2297	3416		
20 O	C.	1550	2.0	21.0	1	1	55	3	10	13.1	1	533	1421	2164	3291	
20 X	C.	1610	2.0	22.0	nil	1	15	3	10	13.1	1	487	1331	2109	3062	
20 G	C.	1560	2.0	21.0	1	1	30	4	50	13.1	1	397	1058	1818	3186	
20 L	C.	1560	2.0	21.0	1	1	10	25	13.1	425	1447	1	1987	3023		
20 M	C.	1620	2.0	21.0	1	1	5	3	0	13.1	1	559	1278	2015	3314	
20 S	C.	1580	2.0	21.0	nil	1	20	3	10	13.1	1	479	1254	1981	3066	
24	C.	1760	2.0	21.0	1	nil	3	20	4	40	13.1	1	475	1376	1872	3186
25	C.	1740	2.0	21.0	1	3	5	4	35	13.1	1	399	1242	1963	3065	

Key : L.L. = low-lime  
 H.L. = high-lime  
 L.L.M.A. = low-lime with MgO and alkalis  
 H.L.M.A. = high-lime with MgO and alkalis  
 C. = derived from commercial clinker

\* Since composition 14 X could not be prevented from dusting, the strength results were abnormally low and are therefore omitted.

TABLE V.—RESISTANCE TO SULPHATE ATTACK.

Cement No.	Composition	Fineness, sq. cm./g.	Time (days) required to expand 1%		Time (days) required for failure	
			5% Sodium sulphate	5% Magnesium sulphate	5% Sodium sulphate	5% Magnesium sulphate
14 X	L.L.	2250	26	20	31	40
14 G	L.L.	2280	37	48	56	68
15 X	L.L.M.A.	2340	24	25	30	30
15 G	L.L.M.A.	2300	35	48	56	68
16 X	L.L.	1410	20	36	28	38
16 G	L.L.	1525	28	40	40	44
17 XA*	H.L.	1590	25	23	26	53
17 GA	H.L.	1700	24	26	27	53
17 XB*	H.L.	1880	21	24	26	52
17 GB	H.L.	1970	33	28	43	47
18 XA	H.L.M.A.	1650	20	22	26	64
18 GA	H.L.M.A.	1630	27	30	44	60
18 XB	H.L.M.A.	2010	25	32	33	42
18 GB	H.L.M.A.	2000	33	52	43	68
19 XA	C.	1580	72	75	230	260
19 GA	C.	1590	>420	>420	>420	>420
19 OA	C.	1640	141	78	179-	260
19 XB	C.	2020	75	105	>380	>380
19 GB	C.	2020	230	191	>420	>420
19 OB	C.	1910	89	62	102	236
19 L	C.	1830	120	130	140	371
19 M	C.	1620	88	65	90	203
19 S	C.	1590	104	306	189	>380
20 X	C.	1610	42	62	53-	125
20 G	C.	1560	70	122	97	>118
20 O	C.	1550	47	55	60-	174
20 L	C.	1560	50	62	81-	105
20 M	C.	1620	30	33	39-	97
20 S	C.	1580	42	38	52-	137
24	C.	1760	83	>150	111	>150
25	C.	1740	37	55	68	>150

A\* indicates fineness 1600 sq. cm. per g.

B\* indicates fineness 2000 sq. cm. per g.

For Key see Table IV.

TABLE VI.—DRYING SHRINKAGE (VOLUME).

Cement No.	Composition	Volume shrinkage %
16 X	L.L.	0.45
16 G	L.L.	0.46
17 XA*	H.L.	0.38
17 GA	H.L.	0.45
17 XB*	H.L.	0.31
17 GB	H.L.	0.31
18 XA	H.L.M.A.	0.34
18 GA	H.L.M.A.	0.36
18 XB	H.L.M.A.	0.38
19 OB	H.L.M.A.	0.35
19 XA	C.	0.35
19 GA	C.	0.35
19 OA	C.	0.35
19 XB	C.	0.40
19 GB	C.	0.40
19 OB	C.	0.41
19 L	C.	0.32
19 M	C.	0.42
19 S	C.	0.47
20 X	C.	0.38
20 G	C.	0.33
20 O	C.	0.34
20 L	C.	0.35
20 M	C.	0.40
20 S	C.	0.40
24	C.	0.34
25	C.	0.36

A\* indicates fineness 1600 sq. cm. per g.

B\* indicates fineness 2000 sq. cm. per g.

The statistical probable error of the determination of volume shrinkage is  $\pm 0.05\%$  or  $\pm 8\%$  of the mean.

For Key see Table IV.

The complete series of the latter is therefore given so that the actual compositions of the different types may be easily compared. Setting time, soundness, strength, and water for normal consistencies are set out for each cement in Table IV, while in Table V the data on chemical resistance in the two sulphate solutions are given, the actual results being quoted in two ways—the number of days elapsing before 1 per cent. expansion took place and the number of days elapsing before complete breakdown of the specimens. Some figures in this table refer to tests which are not yet completed. The data for shrinkage are given in Table VI; the data for heat of hydration are included in Table VII, which gives results for the large-scale tests carried out on cements 24 and 25, where larger quantities were available.

TABLE VII

*Large-scale tests on cements 24 (partly quenched) and 25 (normal product, mainly crystalline)*

Cement No.	Water for normal (neat), %	Setting time				Le Chatelier expansion		
		Initial hr.	min.	Final hr.	min.	Cold, mm.	Hot, mm.	
24	21	4	10	5	20	1	1	
25	22	3	55	6	0	1	1	
Compressive strength, 50 sq. cm. cubes, 1 : 6 concrete, lb. per sq. in.								
Cement w/c, 0·6								
No.	1 day	3 days		7 days		28 days		90 days
24	968	2437		3795		5588		6350
25	963	2188		3535		5272		5967
Temperature rise of concrete stored adiabatically (°C.)*								
Cement No. 1 day 3 days 5 days 7 days								
24	23·6	35·7	40·4	43·7				
25	23·6	33·2	36·9	39·1				

\* Tests on 1 : 6 weights cement: quartz aggregate, w/c, 0·6 (weight).

The heat evolution in calories per g. of cement is 1·9 times the temperature rise.

### DISCUSSION OF RESULTS.

#### *Ease of Grinding of Clinker.*

It may be seen from Table I that for every composition the time of grinding to a given fineness for the quenched preparations exceeded that for the crystalline material. For the two commercial clinkers the hardness of the clinker as received is intermediate between the hardnesses of the annealed and quenched preparations. Also, the addition of magnesia and alkalis to a mix slightly increased the hardness of the clinker, but tended to reduce the difference in hardness between quenched and annealed clinker. At first sight it might seem possible to conclude that advantages in economy of costs of manufacture might be obtained by arranging for more complete crystallisation of the clinker. Tests have been made by other laboratories and works on this same point and, while one investigation confirms the observations,<sup>13</sup> others<sup>14</sup> give completely opposite conclusions. Moreover, it is not certain whether the results in the small ball mill are comparable with those from full-scale mills. The safest conclusion to draw is probably

<sup>13</sup> H. LARMOUR, *Rock Products*, 1934, 37, No. 7, 40.

<sup>14</sup> B. NORDBERG, *ibid.*, 1938, 41, No. 7, 37. O. SWACHHEIM, *Zement*, 1936, 25, 291.

to take the results as an indication that there is some difference in grindability which may perhaps be worth investigating on the full scale.

#### *Setting Time and Soundness.*

On the whole the setting times of the clinkers made in the laboratory were somewhat faster than those of ordinary commercial cements, but apart from this there appeared to be no regular variation of setting time with the mode of preparation of the clinker.

The setting time data given were obtained on the cements immediately after grinding. During subsequent storage in sealed glass bottles some of the cements became flash-setting and others were found to cause stiffening of a mortar while gauging. This stiffening could be worked over and when this was done the strengths, at least at the longer ages, were not affected. On examining the hydration of grains of the cement in an excess of water on microscope slides it was found that those cements which caused stiffening of mortars developed a crystalline hydration product within a few minutes, while the other cements showed no such development of crystals in this short period. When saturated lime water was used in place of water no crystal formation was observed, and, if lime water was used instead of distilled water in the gauging of the mortar, no stiffening occurred. The procedure was therefore adopted of mixing mortars with lime water, whenever a trial showed stiffening to be taking place. No other observations or further tests have been made on this phenomenon except that it was observed only on the "X" type of cement, never on the "G" or "O" types. Not all "X" types were affected.

The flash-setting was different from the stiffening, but no further observations were made on it. It is noted only as a peculiarity affecting some of the "X" cements.

#### *Strength.*

Statistical analysis of the strength results showed that at 1 day the quenched product in three cases gave a higher strength than the annealed product. In two cases the reverse was true, although the differences were smaller, and in five cases there was no significant difference. At 3 and 7 days there were no significant differences, but at 28 days the quenched product gave the higher strength in seven cases, the most notable being No. 15, where the quenched clinker gave a strength 40 per cent. higher than that of the annealed material; No. 18, where the differences were 24 per cent. and 17 per cent. respectively for the cements of 1,600 and 2,000 sq. cm. per g. specific surface; and No. 17 with differences of 22 per cent. and 15 per cent. respectively for the coarse and fine cements.

No case occurred in which the strength of the annealed product exceeded that of the quenched at 28 days, although for four cements the difference was not significant. There was a tendency for the addition of magnesia and alkalis to lower the strength up to 7 days, but the effect was small and not entirely consistent.

The 1-day strength for cements ground to 2,000 sq. cm. per g. exceeded in all cases except one (where there was no significant difference) the strength for the

corresponding cement ground to 1,600 sq. cm. per g. For two compositions this increase in strength on grinding persisted up to 28 days, but with the other compositions the differences at this age were not significant.

Re-burning of commercial clinker produced no significant change in strength, either when the product was quenched or when it was annealed, and there was no significant difference between the cements prepared from the various grain sizes of the commercial clinkers at any age up to 28 days.

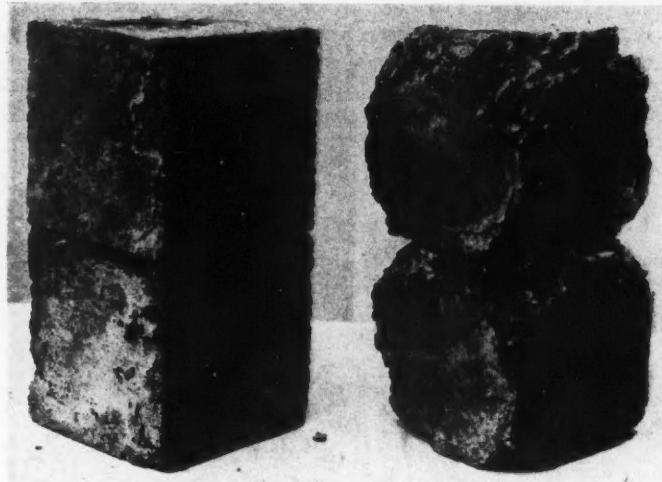
#### *Resistance to Attack by Sulphate Solutions.*

A general difference was observed between the glassy and the crystalline cements in their resistance to attack by sulphate solutions, the former always being superior to the latter—in some cases very markedly ; it will be noted from Table I that composition 17, where there is little change in sulphate-resistance on quenching, was imperfectly quenched. Cements prepared from the commercial clinkers as received were in general intermediate in resistance between those derived from the glassy and the crystalline samples, but were nearer to the latter. In all cases the specimens survived longer in magnesium sulphate than in sodium sulphate. The data on the effect of fineness were not sufficiently complete to make any definite deductions, although the indications were that the effect was small. The effect of magnesia and alkali additions also seemed to be negligible.

Larger tests were made on the cements 24 and 25—those prepared at the kiln—and the conclusions from the small-scale tests were confirmed by the larger. As an example, Plates 1 and 2 show photographs of concretes made from both cements after immersion in the two solutions for 31 weeks. Although disintegration of the crystalline cement (25) is well advanced, the glassy cement (24) is just beginning to show signs of attack.

Further analysis of the results indicated that there was a relation, for the crystalline cements only, between the calculated (Bogue) content of  $3\text{CaO} \cdot \text{Al}_2\text{O}_3$  in a cement and its resistance to sulphate attack. With these cements the resistance to attack by sulphate solutions is increased as the content of crystalline  $3\text{CaO} \cdot \text{Al}_2\text{O}_3$  is reduced. The relation is not linear and there is a tendency at the lower  $3\text{CaO} \cdot \text{Al}_2\text{O}_3$  contents for small reductions in crystalline aluminate to be associated with marked increases in resistance. For the particular compositions examined this effect is most noticeable below 11 per cent. of  $3\text{CaO} \cdot \text{Al}_2\text{O}_3$ , but it would not be safe to assume that 11 per cent. represents a limiting value for all variations of Portland cement composition ; there is, in fact, evidence to the contrary<sup>15</sup> since cements of relatively poor resistance have been found with contents of  $3\text{CaO} \cdot \text{Al}_2\text{O}_3$  as low as 7 per cent. The present series contained compositions all of which had ratios of  $\text{Al}_2\text{O}_3$  to  $\text{Fe}_2\text{O}_3$  of the same order. It is possible that the examination of a wider range might show somewhat different results, as might also cements of wider range of  $\text{CaO}$  to  $\text{SiO}_2$  ratio. Nevertheless, under the restricted conditions of the series, the results do indicate the crystalline

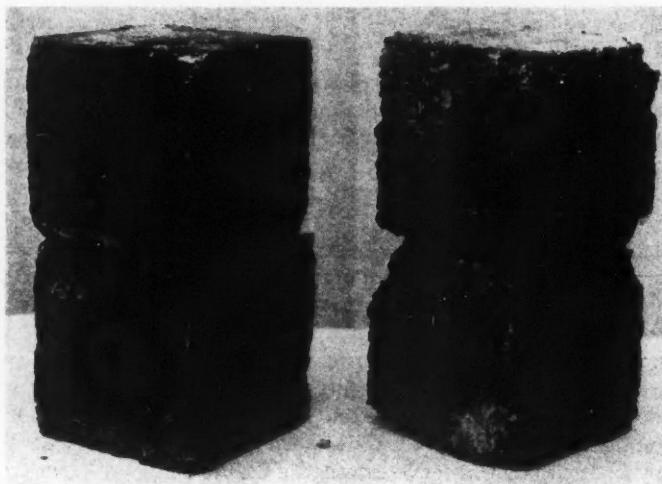
<sup>15</sup> F. M. LEA, *Chem. and Ind.*, 1935, 1522.



Cement 24.

Cement 25.

Plate 1.—Cubes of 1:6 concrete (water-cement ratio 0.6) after 31 weeks' immersion in 5 per cent. sodium sulphate.



Cement 24.

Cement 25.

Plate 2.—Cubes of 1:6 concrete (water-cement ratio 0.6) after 31 weeks' immersion in 5 per cent. magnesium sulphate.

aluminate as being a source of vulnerability to attack of cements by sulphates. Very similar results have been obtained by Gonnerman.<sup>16</sup>

One method for reducing the content of crystalline aluminate is by quicker cooling so that some, at least, of the aluminate is held in the glass, and this, it seems, must be the explanation of the higher resistance of the quenched as compared with the annealed cements. In the case of cement 24 microscopic examination showed it to have been imperfectly quenched, a good deal of crystallisation having taken place in the quenching. The amount of glass formed was apparently

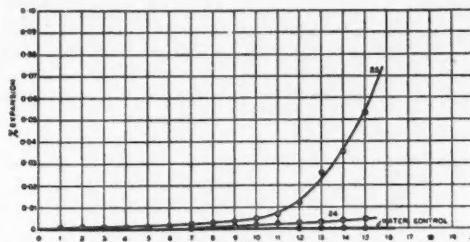


Fig. 1. Expansion of 5-in. rods of 1:3 mortars in 5% sodium sulphate. Cements 24 and 25.

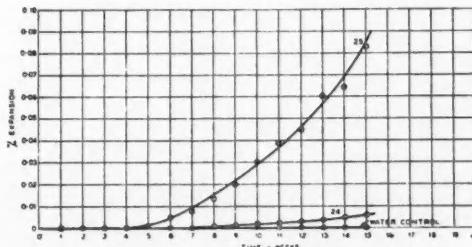


Fig. 2. Expansion of 5-in. rods of 1:3 mortars in 5% magnesium sulphate. Cements 24 and 25.

sufficient to reduce the crystalline aluminate content, however, and as the aluminate content of the normal clinker was already fairly low this reduction was sufficient to increase the resistance very considerably as compared with the normal clinker 25. The full graphs of the change in length with time of large-scale tests on these two cements are given in Figs 1 and 2, as a further demonstration of the difference in resistance obtained.

#### Shrinkage.

As is seen from Table VI, there was no significant difference, within the limits of accuracy of the small-scale method, between the drying shrinkage of cements prepared from quenched and annealed clinker, or between similar cements of different finenesses, or between compositions with and without MgO and alkalis.

<sup>16</sup> H. F. GONNERMAN, *Proc. Amer. Soc. Test. Mat.*, 1934, 34, II.

Larger-scale tests on the shrinkage of prisms of cements 24 and 25 showed, however, a lower shrinkage for the glassy (0·038 per cent.) than for the crystalline cement (0·047 per cent.). The shrinkage of the cement prepared from the  $<\frac{3}{16}$ -in. size of clinker 19 is greater than that for the fraction  $>\frac{3}{16}$ -in.; the differences for other clinker sizes were not significant.

#### *Heat of Hydration.*

The results (Table VII) of the adiabatic calorimeter tests on cements 24 and 25 indicate that the glassy cement has a greater temperature rise on hydration than the crystalline cement after the first day, the increase amounting to about 12 per cent. at 7 days. This is a result which would be expected on theoretical grounds and is confirmed by data recently published by W. Lerch.<sup>17</sup>

#### SUMMARY.

It may be convenient to summarise briefly the points of difference of the two types of cement, expressing these as "advantages" of either type:

- (1) The crystalline cements were easier to grind on small-scale mills, but it is uncertain if this holds true under commercial conditions.
- (2) Glassy cements gave rise to no difficulty in controlling setting time, whereas some trouble was experienced with the crystalline cements.
- (3) The crystalline cements had the lower heat of hydration.
- (4) The glassy cements gave somewhat higher strengths at an age of 28 days.
- (5) There is some little evidence that the drying shrinkage of glassy cements may be lower than that of the crystalline cements.
- (6) The glassy cements had a considerably higher resistance to attack by sulphate solutions than the crystalline cements.

<sup>17</sup> *J. Res. Nat. Bur. Stand.*, 1938, 21, 235.

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#### New White Cement Plant.

A SMALL plant has been erected at Carrara, U.S.A., for the production of white cement, using white marble limestone and white silica-aluminium clay. If these raw materials prove satisfactory it is proposed to erect a plant with an initial capacity of 1,000 barrels a day. The new works is operated by the Carneva Portland Cement Co.

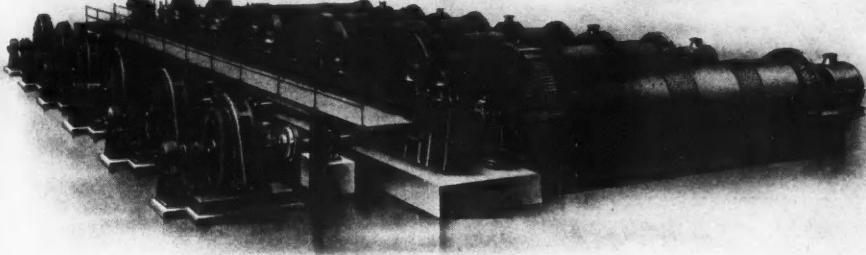
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